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INVESTIGATIONS LEADING TO THE DEVELOPMENT
OF A PRIMARY ZINC-SILVER OXIDE BATTERY
OF IMPROVED PERFORMANCE CHARACTERISTICS
SUMMARY REPORT NO. 2
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I. PURPOSE

The purpose of this contract is to generate design data making possible the construction of a reliable primary zinc-silver oxide battery of improved activated charge retention characteristics, greater voltage control, high energy density, increased temperature stability, and reduced gassing characteristics.

II. ABSTRACT

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A

A series of experimental cells have been subjected to a statistical design study using a fractional factorial main-effect plan. Several significant cell construction variables have been isolated by use of the half-normal plot. It is suggested that further studies be initiated to discover the optimum combination of these variables. A computer routine is being used to determine an equation describing capacity and voltage responses for cells tested in the recently concluded experimental study.

AUTHOR

III. FACTUAL DATA AND DISCUSSION

A. Fractional Factorial Study

1. Analysis by Inspection of Data

It was anticipated that many cell construction variables might affect performance characteristics. (Several of these were selected for a study to evaluate the effects of these factors and to allow the design of a primary zinc - silver oxide cell of improved performance characteristics.) Factors designated for study in this experimental series are revealed by Table No. X.

It was decided that a maximum amount of data could be accumulated by use of statistical methods. Therefore, a basic main-effect plan was selected from Addelman's Orthogonal Main-Effect Plans. This plan permitted the construction of a set of twenty-seven (27) cell types following a fractional factorial pattern. Combinations of cell construction variables suggested by this plan were tested as shown in Table No. XI.

Materials were obtained and cells constructed for five duplicate sets of cells. However, it proved impractical to electroform "pasted" zinc negative material by an experimental process within the precise controls required for a sensitive experiment.

Attempts had not been made previously to adapt this method of negative material formulation to use in the primary zinc - silver oxide system. Cells involving this level of Factor "H" were not constructed, thus reducing the number of test cells in each set to eighteen (18), which imparted a degree of "bias" to the experiments.

Cells were constructed to deliver 10 to 12 ampere-hours, initially, at the nominal 20-minute rate. In order to expedite accumulation of data, cells were activated with 22 cc of the appropriate electrolyte and placed on charged stand at +130° F. Sets of cells were removed from stand periodically and allowed to cool to +75° F, then discharged at a 30-ampere rate to an individual cell end voltage of 1.10 volts. Discharge voltages were monitored closely to allow evaluation of cell performance characteristics. Discharges were conducted after activated stand periods of 2 hours, 4 days, 6 days, 8 days and 10 days at +130° F.

Both a voltage and a capacity response were obtained for each cell resulting from its discharge characteristics. The capacity response is the number of minutes during which the cell terminal voltage exceeded 1.10 volts.

TABLE NO. X
TEST VARIABLES AND THEIR LEVELS

A. Additives to Electrolyte

A_0 - None
 A_1 - 1% gel
 A_2 - MnO (at saturation)
 A_3 - LiOH (at saturation)

B. Electrolyte Concentration

B_0 - 35%
 B_1 - 40%
 B_2 - 45%

C. Positive Material Density (gms./cu.in.)

C_0 - 68
 C_1 - 74
 C_2 - 80

D. Positive Grid Metal

D_0 - 4/0 Ni
 D_1 - 4/0 Ag

E. Negative Material Density

E_0 - 40
 E_1 - 45
 E_2 - 50

F. Additive Content in Negative Plate*

F_0 - 1%
 F_1 - 2%
 F_2 - 4%

G. Negative Grid Metal

G_0 - Copper (4/0)
 G_1 - Silver Flashed Cu (4/0)
 G_2 - Silver (4/0)

H. Negative Formulation **

H_0 - Pasted ***
 H_1 - Sponge
 H_2 - Metallic

* The nature of this additive and its introduction into the negative plate is proprietary.

** These are descriptive names referring to three types of negative plates prepared by proprietary procedures.

*** Cells involving this level were not constructed.

TABLE NO. XI
FRACTIONAL FACTORIAL PATTERN
TWENTY-SEVEN CELL GROUP

CELL NO.	LEVELS							
	A	B	C	D	E	F	G	H
1	0	0	0	0	0	0	0	0
2	0	1	1	1	2	1	2	2
3	0	2	2	0	1	2	1	1
4	1	0	0	0	0	1	1	1
5	1	1	1	0	2	2	0	0
6	1	2	2	1	1	0	2	2
7	2	0	0	1	0	2	2	2
8	2	1	1	0	2	0	1	1
9	2	2	2	0	1	1	0	0
10	1	0	1	1	1	0	0	1
11	1	1	2	0	0	1	2	0
12	1	2	0	0	2	2	1	2
13	3	0	1	0	1	1	1	2
14	3	1	2	1	0	2	0	1
15	3	2	0	0	2	0	2	0
16	3	0	1	0	1	2	2	0
17	3	1	2	0	0	0	1	2
18	3	2	0	1	2	1	0	1
19	2	0	2	0	2	0	0	2
20	2	1	0	0	1	1	2	1
21	2	2	1	1	0	2	1	0
22	2	0	2	1	2	1	1	0
23	2	1	0	0	1	2	0	2
24	2	2	1	0	0	0	2	1
25	0	0	2	0	2	2	2	1
26	0	1	0	1	1	0	1	0
27	0	2	1	0	0	1	0	2

The voltage response, expressed as a percentage, was obtained by dividing the cell voltage after 15 seconds of discharge by 1.50 volts (an arbitrary value), and multiplying the result by 100. This response reflects both voltage magnitude and voltage control.

Cell responses for the respective stand periods are exhibited by Table Nos. XII and XIII.

Data have been treated by several methods to gain maximum information. The first of these consisted of calculating mean capacities for cells involving each factor level. This resulted in the values exhibited in Table Nos. XIV through XVII. Factors in which consistent trends appeared are "B", "D", "E" and "H". This indicates that capacity retention is improved through use of 45% KOH electrolyte by weight, nickel positive grid, negative material of 40 grams per cubic inch apparent density, and negative material of "sponge" crystalline structure.

TABLE NO. XII
RESPONSES (voltage**)

CELL NO.	2 hr.	4 da.	6 da.	8 da.	10 da.
2	96.0	86.0	85.3	84.0	84.0
3	88.7	78.7	81.3	73.3	76.0
4	92.7	86.0	85.0*	84.0	84.7
6	94.7	89.3	86.0	80.7	82.7
7	96.0	92.0	88.0	85.3	88.7
8	90.7	79.3	82.0	78.7	83.3
10	96.0	92.0	92.0	88.0	89.3
12	90.7	76.0	76.0	74.0	79.3
13	90.7	84.7	85.3	82.0	86.0
14	94.7	95.3	92.0	89.3	89.3
17	87.3	77.3	82.0	73.3	82.0
18	93.3	85.3	85.3	74.7	77.3
19	90.7	88.0*	84.0	80.0*	77.3
20	91.3	84.0*	81.3	77.3	82.0
23	92.0	80.7	85.3	80.0	82.7
24	89.3	76.0	80.7	75.3	79.3
25	92.0	84.7	86.7	84.0*	82.0*
27	90.7	79.3	79.3	76.3	75.0*

* Responses for these cells (which failed to deliver usable capacity) were estimated from the response pattern.

** These responses are obtained by dividing the cell discharge voltage after 15 seconds by 1.50 volts. This artificial response facilitates handling of data.

TABLE NO. XIII
CELL RESPONSES (capacity)
 (Discharge capacity in minutes at a 30-ampere rate)

CELL NO.	2 hr.	4 da.	6 da.	8 da.	10 da.
2	19.0	13.0	12.25	11.0	9.0
3	30.0	28.0	23.00	21.0	18.0
4	27.0	24.0	21.00**	19.0	19.5
6	24.0	20.0	19.5	17.0	19.0
7	22.0	17.0	13.75	14.0	15.0
8	29.0	23.0	22.50	18.0	17.0
10	24.0	22.0	21.20	20.0	18.0
12	28.5	23.0	19.25	15.5	19.0
13	22.0	13.0	17.30	9.0	9.0
14	25.5	21.0	20.75	20.0	18.0
17	27.0	25.0	21.50	19.0	17.0
18	24.0	19.0	19.75	17.0	16.0
19	23.0	20.0*	11.50	14.0*	19.0
20	30.5	24.0*	19.75	19.0	20.0
23	25.0	28.0	18.50	17.0	17.0
24	28.0	25.0	24.50	22.5	22.0
25	24.0	13.0	15.50	13.0*	12.0*
27	30.0	21.0	17.50	20.0	17.0*

* These are estimated responses based upon trends in cell performance characteristics.

TABLE NO. XIV
MEAN CAPACITY IN MINUTES
 (30-ampere rate)
 (2 hr. stand)

FACTOR	LEVEL			
	0	1	2	3
A	25.8	26.0	26.3	24.6
B	23.5	26.0	27.4	
C	26.1	25.3	25.6	
D	27.0	23.1	--	
E	26.6	25.9	24.6	
F	25.8	24.8	25.8	
G	25.3	27.3	24.6	
H		26.9	24.5	

TABLE NO. XV
MEAN CAPACITY IN MINUTES
(30-ampere rate)
(6 da. stand)

FACTOR	<u>L E V E L</u>			
	0	1	2	3
A	17.1	20.0	18.6	19.8
B	15.1	19.2	20.6	
C	18.2	19.2	18.6	
D	19.2	17.9		
E	19.6	19.9	16.8	
F	20.1	17.3	18.9	
G	18.2	20.6	17.5	
H	--	20.9	16.8	

TABLE NO. XVI
MEAN CAPACITY IN MINUTES
(30-ampere rate)
(8 da. stand)

FACTOR	<u>L E V E L</u>			
	0	1	2	3
A	17.1	17.9	18.1	16.2
B	17.1	17.3	18.7	
C	16.9	16.0	19.2	
D	16.6	16.5		
E	19.1	17.2	15.4	
F	19.3	15.8	17.5	
G	17.9	16.9	16.7	
H	--	19.6	15.3	

TABLE NO. XVII
MEAN CAPACITY IN MINUTES
(30-ampere rate)
(10 da. stand)

FACTOR	<u>L E V E L</u>			
	0	1	2	3
A	13.5	18.9	18.5	15.0
B	16.1	16.3	18.8	
C	17.7	15.0	18.2	
D	17.8	15.8		
E	18.3	16.8	16.0	
F	18.7	14.7	17.4	
G	17.2	16.6	17.0	
H	--	18.9	15.5	

2. Analysis by Half-Normal Plots

Following this preliminary analysis, effort was made to interpret the data by means of half-normal plots. Fifteen comparisons were selected for study and assigned weighted values as revealed in Table No. XVIII. These comparisons are linear, quadratic, or cubic as indicated by the subscripts, L, Q, or C, respectively.

Arithmetic calculations involved in this analysis proceeded as follows:

1. $\sqrt{\sum c^2}$ was taken for the corresponding comparison from Table No. XVIII.

2. ΣcX was obtained by multiplying the response of each cell in the group by the value associated with that comparison in Table No. XVIII, then computing the sum of these terms.

3. The contrast value is $\frac{\Sigma cX}{\sqrt{\sum c^2}}$. The algebraic sign associated with the contrast value is not used in preparing half-normal plots, but indicates which level or combination of levels in a comparison is more desirable.

4. Contrast values were arranged in order of magnitude and plotted on half-normal paper as per a 15 order-number sample. Points falling away from the half-normal line were judged to be significant.

TABLE NO. XVIII
FACTOR LEVELS AND COMPARISONS

NO.	FACTOR LEVELS								COMPARISONS								
	A	B	C	D	E	F	G	H	A _L	A _Q	B _L	B _Q	C _L	C _Q	D _L	D _Q	
2	0	1	1	2	1	2	2	-3	-1	1	0	2	2	1	-1	0	2
3	0	2	2	0	1	2	1	-3	-1	1	-1	-1	-1	0	2	-1	0
4	1	0	0	0	1	1	1	-1	-1	-1	-1	-1	-1	0	2	0	2
6	1	2	2	1	1	0	2	2	-1	1	-1	1	-1	2	-1	-1	1
7	2	0	0	1	0	2	2	1	-3	-1	-1	-1	2	-1	-1	1	-1
8	2	1	1	0	2	0	1	1	-3	-1	0	2	-1	1	-1	0	2
10	1	0	1	1	0	0	1	-1	3	-1	-1	0	2	2	-1	-1	-1
12	1	2	0	0	2	1	2	-1	3	-1	-1	-1	-1	1	-1	0	2
13	3	0	1	0	1	1	2	3	1	1	-1	0	2	-1	1	0	2
14	3	1	2	1	0	2	0	1	3	1	0	2	-1	2	0	2	1
17	3	1	2	0	0	0	1	2	3	1	1	0	2	-1	1	0	2
18	3	2	0	1	2	1	0	1	3	1	1	-1	-1	2	1	-1	-1
19	2	0	2	0	0	2	1	-3	-1	-1	-1	0	2	-1	1	-1	-1
20	2	1	0	0	1	1	2	1	-3	-1	0	2	-1	1	0	2	1
23	2	1	0	0	1	2	0	2	1	-3	-1	0	2	-1	1	-1	-1
24	2	2	1	0	0	0	2	1	-3	-1	1	-1	0	2	-1	-1	-1
25	0	0	2	0	2	2	1	-3	-1	1	-1	1	-1	1	-1	1	-1
27	0	2	1	0	0	1	0	-3	-1	1	-1	0	2	-1	1	-1	1
	Σc^2	82	98	18	12	36	12	36	36	12	36	12	36	12	36	18	
	$\sqrt{\Sigma c^2}$	9.05	9.90	4.24	3.464	6.0	3.464	6.0	6.0	3.464	6.0	3.464	6.0	3.464	6.0	4.24	

These calculations were carried out for both voltage and capacity responses, and for varying periods of activated stand times. Results of this procedure are exhibited by Table Nos. XIX and XX. Data in these tables reveal no clearly consistent trends, but do indicate significance of some comparisons.

In these first attempts at statistical analysis of data, a "zero" value was used as a response for all cells which had shorted internally and had not delivered capacity. This caused bias in several comparisons, since the comparisons involving electrolyte concentration and composition are those primarily expected to hasten failure of the separator systems and resultant electrical shorting. Zero responses, then, did not reflect progressive deterioration of cell capacity due to electrochemical changes within the body of the active materials.

For this reason, responses were estimated which might have exhibited had not separator failure occurred. This was possible because of consistent decreases in cell responses with increased activated stand times.

All significance calculations were then repeated. Contrast values which were obtained are revealed by Table Nos. XXI and XXII. Half-normal plots compiled using these response values are exhibited by Figure Nos. 20 through 26.

Plots involving capacity retention are less informative than the accumulated contrast values listed in Table No. XXI. Apparently significant effects relative to capacity are A_L , A_C , A_Q , B_L , D , E_L , F_L , F_Q , G_L and H .

Significant effects with respect to voltage are listed in Table No. XXIV.

TABLE NO. XIX
PRELIMINARY SIGNIFICANCE ESTIMATIONS

EFFECT	STAND PERIOD		
	2 hours	6 days at +130° F	8 days at +130° F
A_C (no additive & MnO_2)	5%	---	---
A_L (A_3)	---	5%	---
B_L	---	---	5%
A_Q (no additive & $LiOH$)	5%	---	---
F_L	---	5%	---
E_L (E_0)	---	---	5%

TABLE NO. XX
PRELIMINARY SIGNIFICANCE ESTIMATIONS

EFFECT	STAND PERIOD				
	2 hours	4 days	6 days	8 days	10 days
A _C (A ₀ , A ₂)	1%	---	1%	---	---
A _L (A ₃)	5%	---	---	5%	1%
A _Q (A ₀ , A ₃)	5%	---	---	5%	1%
D (D ₀)	---	5%	---	---	---
E _L (E ₀)	---	5%	---	5%	---
F _L (F ₀)	5%	---	---	---	---

TABLE NO. XXI
CONTRAST VALUES
 (with respect to capacity)

EFFECT	STAND PERIOD				
	2 hours	4 days	6 days	8 days	10 days
A _L	4.5	6.1	6.8	3.7	2.6
A _C	-18.1	-13.6	-7.8	-10.0	-10.1
A _Q	-14.0	-16.7	-6.6	-10.7	-16.4
B _L	6.5	2.0	-12.9	6.9	5.3
B _Q	0.9	3.2	1.2	1.0	-1.3
C _L	1.0	-1.7	0.2	0.7	-1.0
C _Q	-1.1	-4.3	1.1	0.8	-4.3
D	-7.8	-6.8	-7.1	-2.3	-2.8
E _L	-3.3	-6.4	-12.6	-8.8	-4.8
E _Q	0.7	-3.7	3.1	0.5	-1.3
F _L	-8.7	-1.4	-7.9	-8.7	-3.8
F _Q	-0.8	-6.8	-2.1	-3.5	-5.0
G _L	-5.2	-6.1	-1.1	-3.3	-0.6
G _Q	4.7	5.2	-5.8	-0.3	-0.9
H	-5.1	-4.0	-8.7	-7.4	-2.5

TABLE NO. XXII
CONTRAST VALUES
 (with respect to voltage)

EFFECT	STAND PERIOD				
	2 hours	4 days	6 days	8 days	10 days
A _L	19.0	21.9	21.9	34.0	24.3
A _C	-56.1	-46.1	-48.0	-45.2	-35.8
A _Q	-21.2	-40.9	-38.9	-39.3	-41.9
B _L	4.7	-12.4	-6.5	-14.2	-11.1
B _Q	0.3	-1.1	16.6	1.3	4.8
C _L	-2.3	19.8	3.2	44.3	-1.6
C _Q	0.5	-3.9	0.6	2.1	13.0
D	9.1	17.5	24.6	11.0	8.8
E _L	0.8	-27.5	2.2	-2.3	-4.6
E _Q	0.4	2.27	2.7	9.6	2.5
F _L	-24.6	7.2	-22.1	-19.1	1.2
F _Q	1.1	0.1	-2.2	-0.9	-2.3
G _L	0.5	-26.9	-2.9	-0.4	2.3
G _Q	-5.9	-13.1	-7.1	-7.4	11.4
H	0.0	-1.9	-3.6	-2.2	-1.3

TABLE NO. XXIII
SIGNIFICANT EFFECTS
 (with respect to voltage)

2 hours	4 days	6 days	8 days	10 days
A _C	A _C	A _C	A _C	A _Q
F _L	A _Q	A _Q	C _L	A _C
A _Q	E _L	D	A _Q	A _L
A _L	G _L	F _L	A _L	C _Q
D	A _L	A _L	F _L	G _Q
G _Q	C _L	B _Q	B _L	B _L
B _L	D	G _Q	D	D
C _L	G _Q	B _L	E _Q	B _Q
	F _L		G _Q	C _L

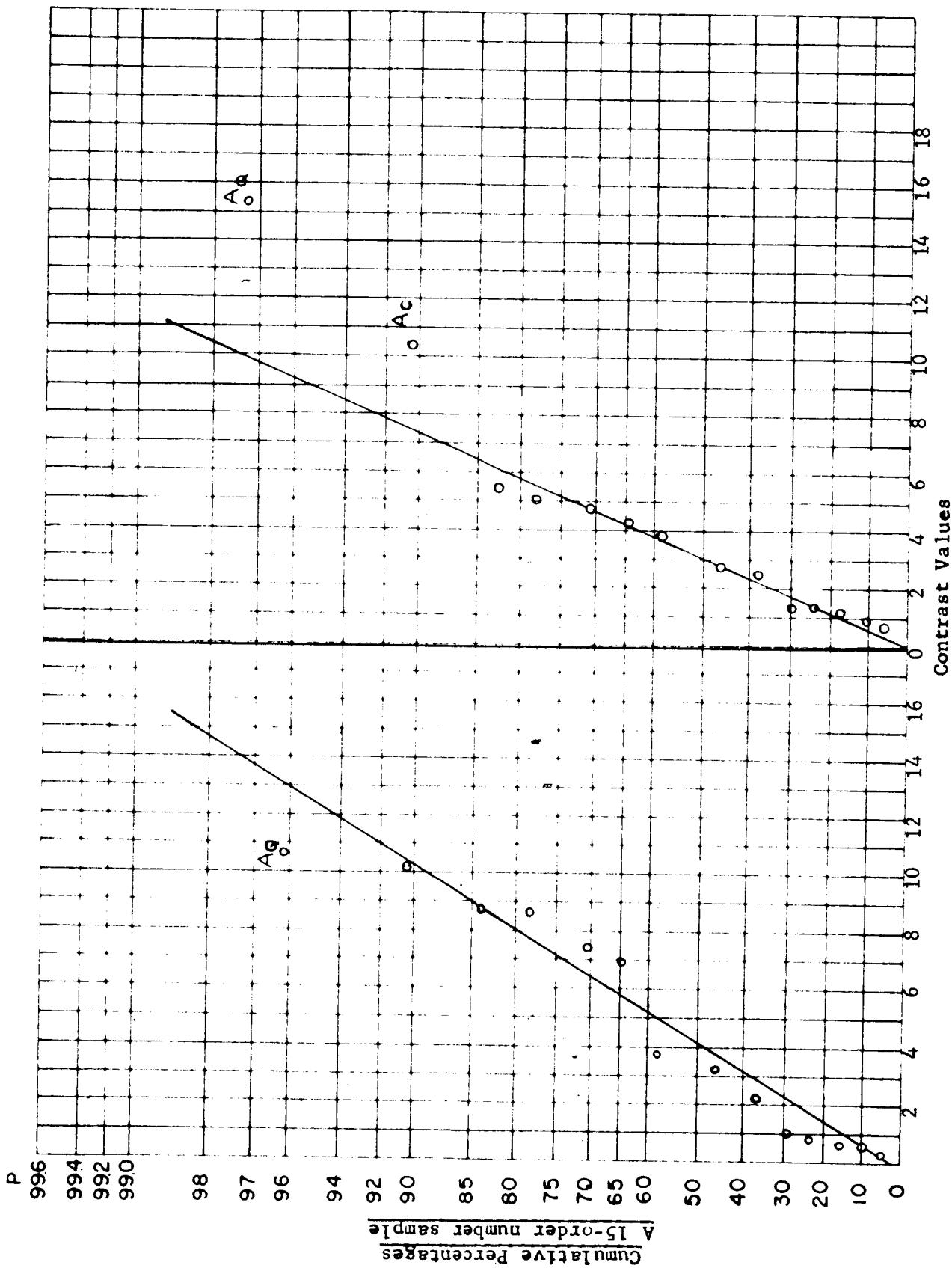


FIGURE NO. 20
(8-day stand)

FIGURE NO. 21
(10-day stand)

SIGNIFICANCE OF EFFECTS
WITH RESPECT TO CAPACITY

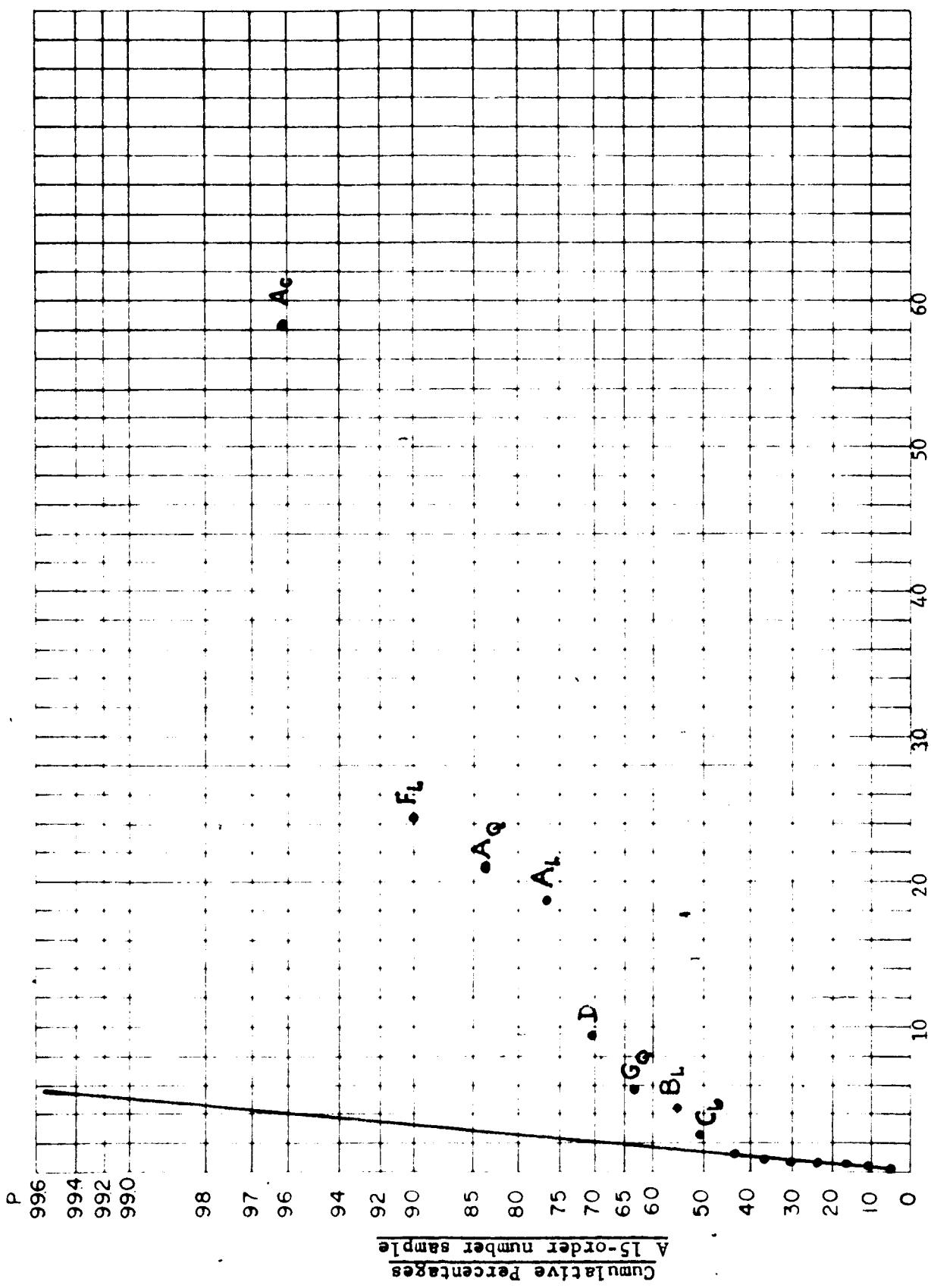


FIGURE NO. 22
SIGNIFICANCE OF EFFECTS
WITH RESPECT TO VOLTAGE
2-HOUR STAND

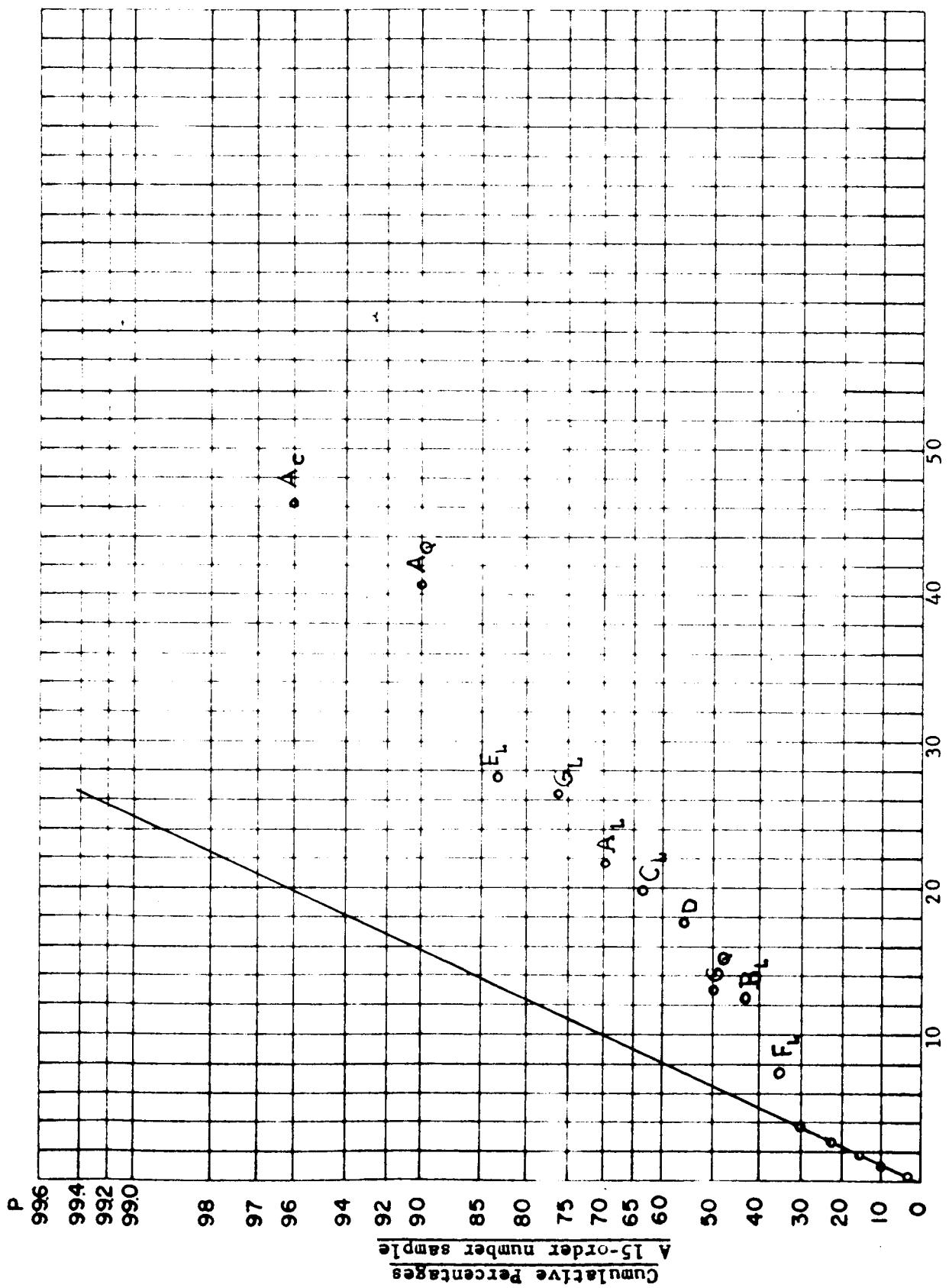


FIGURE NO. 23
SIGNIFICANCE OF EFFECTS
WITH RESPECT TO VOLTAGE
4 - DAY STAND

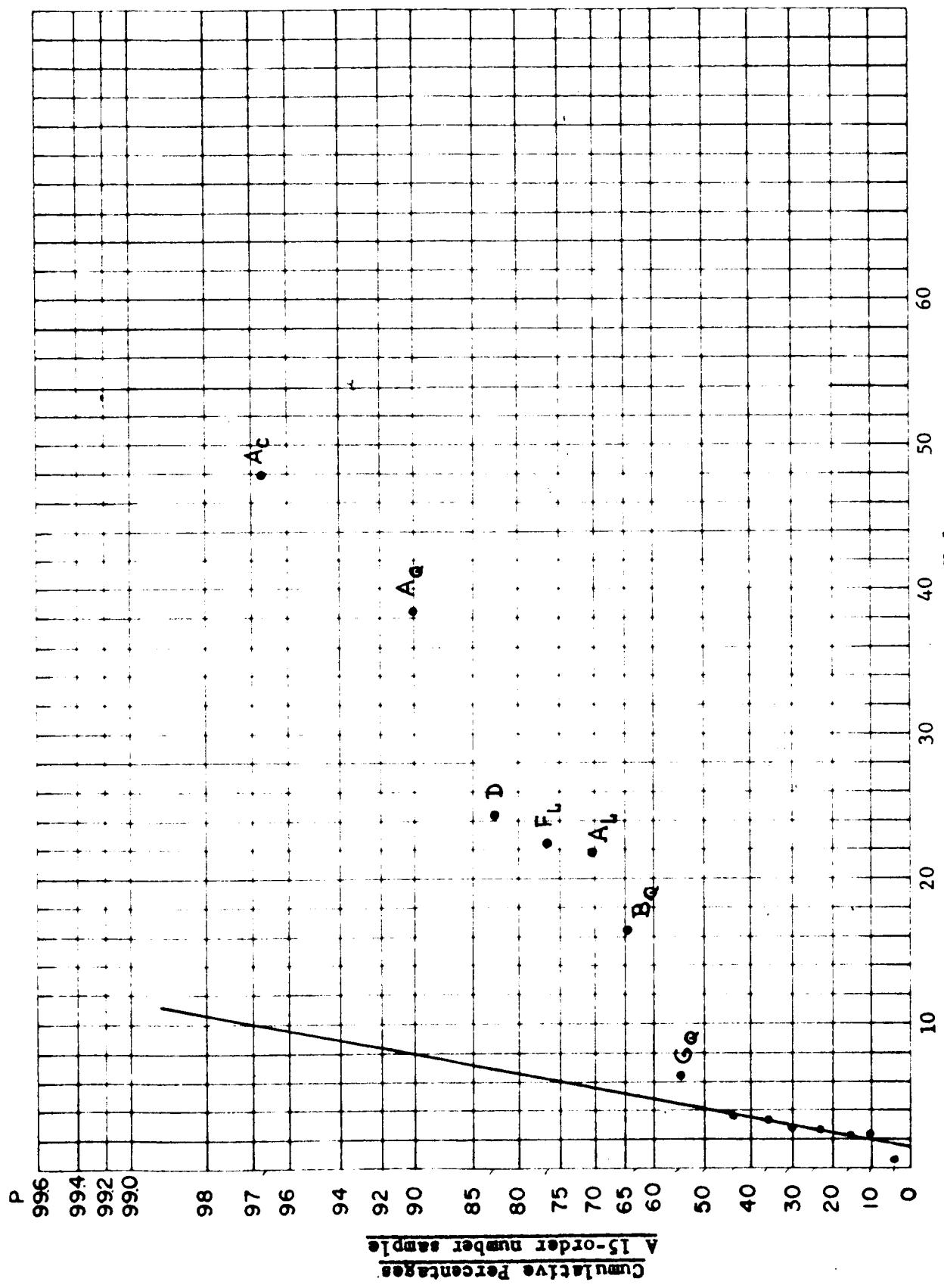


FIGURE NO. 24
SIGNIFICANCE OF EFFECTS
WITH RESPECT TO VOLTAGE
6-DAY STAND

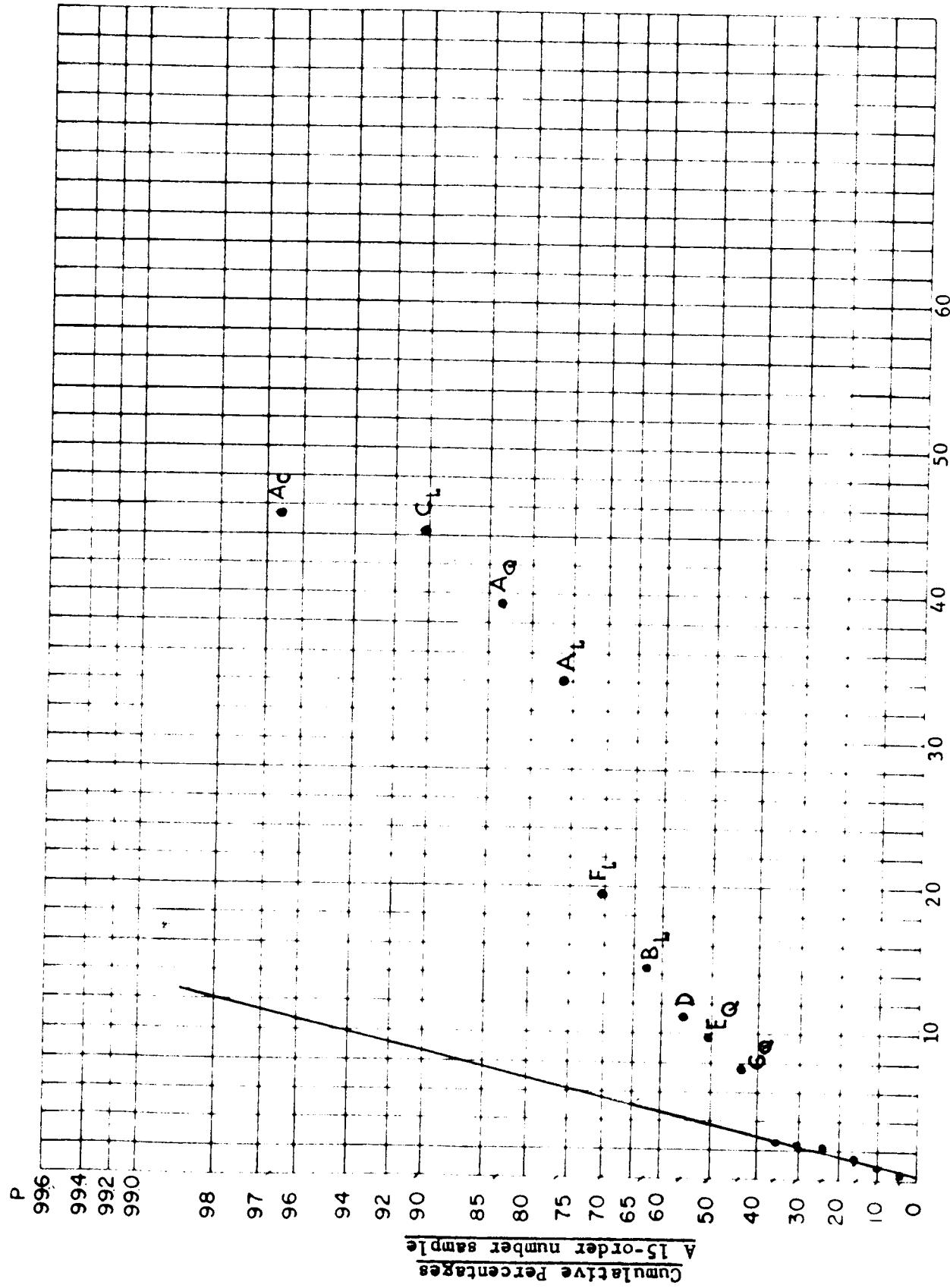


FIGURE NO. 25
SIGNIFICANCE OF EFFECTS
WITH RESPECT TO VOLTAGE
8-DAY STAND

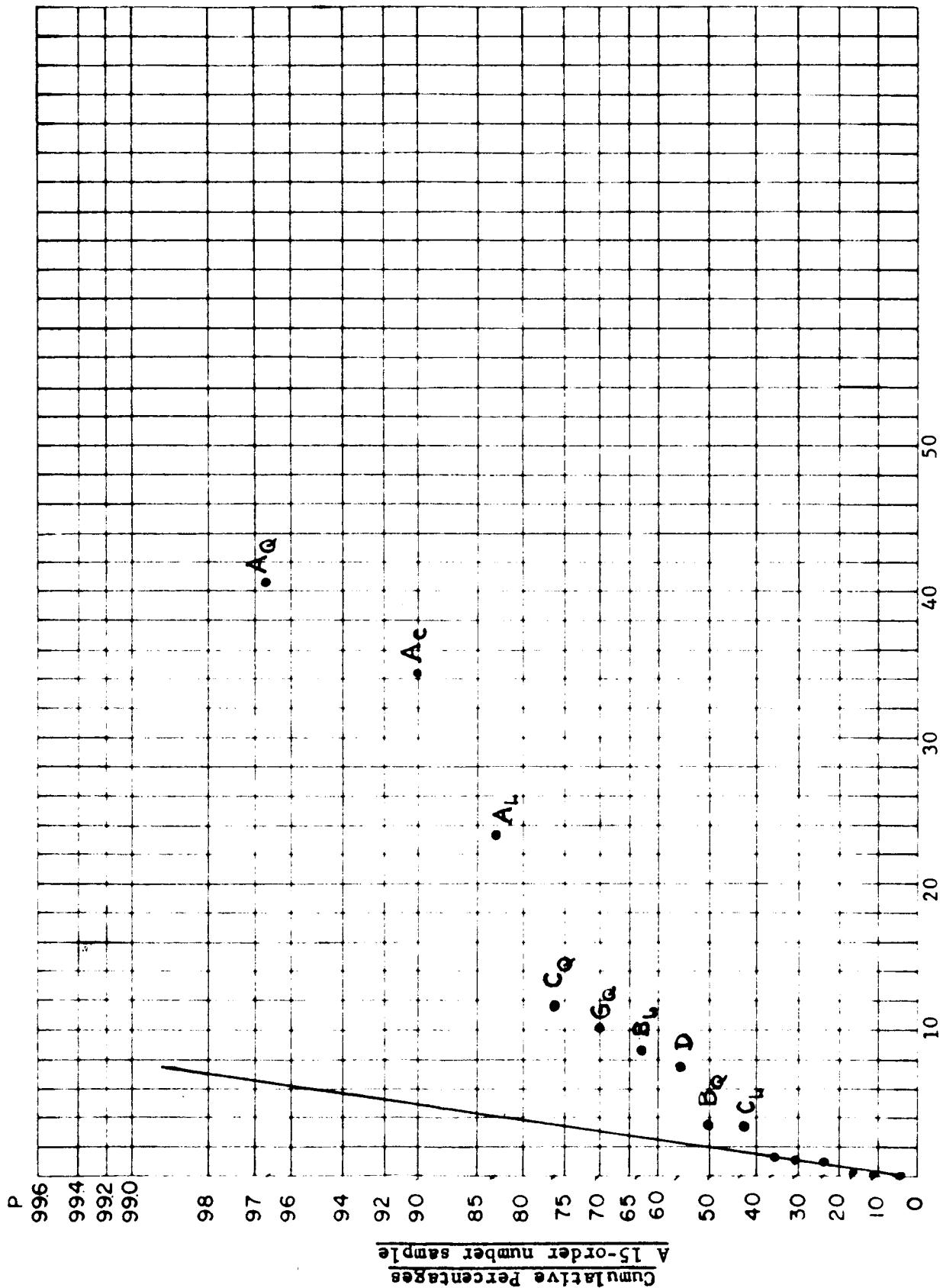


FIGURE NO. 26
SIGNIFICANCE OF EFFECTS
WITH RESPECT TO VOLTAGE
10-DAY STAND

Apparent significance of comparisons relating to Factor "A" stems from non-orthogonality of the plan. Six cells were tested, which included level " A_2 ", while the remaining levels were used in only four cells each, thus causing bias toward the " A_2 " level. Table No. XXIV reveals mean responses for cells involving each level of Factor "A" for a stand period of six days.

TABLE NO. XXIV
EFFECT OF FACTOR "A", ELECTROLYTE ADDITIVES
(6 days stand)

LEVEL OF "A"	VOLTAGE RESPONSE	CAPACITY RESPONSE
A_0 , no ad- ditive	82.3	17.1
A_1 , gel.	84.8	20.3
A_2 , MnO_2	84.9	18.4
A_3 , LiOH	86.1	19.3

Voltage data indicate possible beneficial results from the addition of LiOH (A_3) to the electrolyte, but are otherwise inconclusive within experimental error.

It has become obvious that care must be used in interpreting half-normal plots. These plots are predicated on the assumption of several "zero" effect or insignificant comparisons through which points the half-normal line is drawn. In a plan which has been designed to evaluate factors and comparisons anticipated to be significant, the position of the half-normal line may be determined by a very few points. This makes the exact location difficult to ascertain and reduces confidence in the results.

3. Curve-Fitting by Computer

Another method of data analysis consists of assuming linear, quadratic and cubic effects, and determining an equation which "fits" the response data. This is a regression routine best accomplished by use of standard computer regression programs. Both voltage and capacity data are being so treated by Dr. Leroy Folks of the Department of Mathematics and Statistics of Oklahoma State University. Data are being fitted to an equation of the type:

$$\text{Response} = \mu + a_1 + \theta_1 b + \theta_2 b^2 + \gamma C + \delta_2 C^2 + \dots ,$$

where one or more terms are used for each factor and coefficients are determined by the computer regression routine. This allows determination of an equation describing both voltage and capacity for each activated stand time. Calculations will be made to determine how well this equation describes the actual data in each case.

B. Pre-Prototype Cell Construction

A group of preliminary pre-prototype cells has been constructed and put on activated stand. These were constructed primarily to evaluate the relative abilities of silver and nickel positive grids to maintain cell capacity associated with the zinc - divalent silver oxide couple. This test series, nearly complete, will be included in the 3rd Summary Report.

IV. CONCLUSIONS

1. Half-normal plots, if interpreted correctly, yield a conservative estimate of significance of main effects in fractional factorial experiments.
2. Several variables have been revealed to be significant in their effects upon cell performance characteristics. These are as follows: additives to the electrolyte, electrolyte concentration, positive and negative grid metals, negative material density, additives to the negative material, and method of formulation of the negative material.
3. Sufficient data have been obtained to design a pre-prototype primary zinc - silver oxide cell of improved characteristics.

V. PROGRAM FOR THE NEXT INTERVAL

1. Equations for capacity and voltage responses with respect to cell construction variables will be obtained and tested to see how well they describe experimental data.
2. Data will be obtained relative to the abilities of silver and nickel positive grids to maintain voltage and capacity on stand when utilizing active material at the divalent silver oxide level.
3. A final cell of improved characteristics will be design and constructed, utilizing the results of fractional factorial studies.

VI. PERSONNEL

The following totals of man-hours have been expended during the contract period:

Engineering	- 1078 hours
Technical	- <u>2195 hours</u>
TOTAL	3273 hours

BRH/bk

A P P E N D I X

MASTER SCHEDULE

PROGRAM PRIMARY ZINC-SILVER OXIDE BATTERY
 CONTRACT NAS 8-5493
 REFERENCE TA-242

1 July 1963
 ← P.O. DATE

PREPARED *Bill R. Hartman*
 APPROVED *J. T. D.*
 APPROVED *C. M. M.*

ACTIVITIES												MILESTONES												
1963						1964																		
SEPARATOR STUDIES																								
Open Types																								
Closed Types																								
Special Types																								
ACTIVE MATERIAL FORMULATION																								
Positive Material																								
Negative Material																								
PLATE CONSTRUCTION AND ELEMENT DESIGN																								
Spongy vs. Metallic Zinc																								
Dry Charged Spongy Zinc																								
Grid Materials																								
ELECTROLYTE																								
Effect of Concentration																								
Additives to Electrolyte																								
CELL AND BATTERY CONSTRUCTION																								
Electrolyte Retention																								
Voltage Regulation																								
Energy Density																								
Thermal Characteristics																								
Evaluation of State-of-Art																								
REACTION MECHANISMS																								
Theoretical Investigation																								
Gassing Rate																								
RETENTION EVALUATION																								
PROGRESS REPORTS	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*
AFTER GO-AHEAD	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
CALENDAR	I	II	III	IV	V	VI	VII	VIII	IX	X	XI	XII	JULY	AUGUST	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MARCH	APRIL	MAY	JUNE

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